भारतीय मानक
Indian Standard

IS 2741 : 2022

β-नेफ़थाल — विशिष्टि

(*तीसरा पुनरीक्षण*)

β-Napthol — Specification

(*Third Revision*)

ICS 71.080.90

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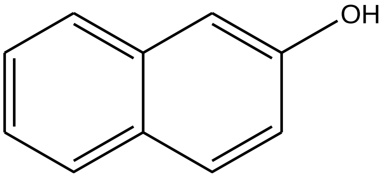
July 2022 **Price Group 7**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

β-Naphthol is an important raw material in the dyestuff industry. It is used in the manufacture of pigments and its derivatives are used in the manufacture of azo dyes. It is also known as 2-hydroxy-naphthalene and is represented by the following structural formula:



β-NAPHTHOL

IUPAC Name: Naphthalen-2-ol
[MOLECULAR MASS: 144]
(CAS REGISTRY NO.:135-19-3)

This standard was first published in 1964 and subsequently revised in 1973 and 1989. The first revision was carried out to update the standard in accordance with the quality of the material being produced at that time and also to introduce thin layer chromatographic method for estimation of *α*-naphthol content, which is the major impurity in *β*-naphthol. The alternate method for this purpose, namely, colorimetric method using sodium cupricyanide in alkaline solution was also substituted by a method using ferric chloride solution, chloroform or carbon disulphide.

In the second revision, iodimetric method for assay was introduced as an alternate method to coupling value method given in **14** of IS 5299 'Methods of sampling and tests for dye intermediates'.

Considering the development in latest analytical techniques in the last 15 years, committee decided to revise this standard. In this (third) revision requirement for naphthalene content and purity by HPLC method have been incorporated. The test method for determination of *α*-naphthol has also been updated to HPLC method

The composition of the Committee responsible for formulation of this standard is given in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 :2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

β-NAPHTHOL — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for β-naphthol.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of dangerous goods Part 1 Dangerous goods
IS 2552 : 1989	Steel Drums (galvanized and ungalvanized) — Specification (*third revision*)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless to pale brown powder or scales. It shall be free from dust and other visible impurities.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 and col 5 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable paper bags, plywood boxes or drums (*conforming to* IS 2552).

4.2 Marking

4.2.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material;
d) Batch or lot number;
e) Month and year of the manufacturer; and
f) Any other statutory requirement.

4.2.2 Each container shall bear the minimum cautionary notice worded as under:

ACUTE IRRITANT. AVOID INHALATION AND CONTACT WITH SKIN. HANDLE WITH CARE. KEEP PROTECTED FROM HEAT. KEEP AWAY FROM FOODSTUFFS AND ANIMAL FEEDS

4.2.3 The containers shall also be suitably labelled with the symbol of 'Danger of Poisoning' [*see* IS 1260 (Part 1)].

4.2.4 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the

Bureau of Indian Standards Act, 2016 and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

Table 1 Requirements for β-Naphthol

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Annex	Clause No. in IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Crystallizing point, °C, *Min*	120	—	**8**
ii)	Purity by coupling value, percent by mass, *Min*	98.5	A	—
	Or			
	Purity by HPLC[1], percent by mass, *Min*	98.5	B	—
iii)	Insoluble in dilute sodium hydroxide solution, percent by mass	0.2	—	**11.2**
iv)	Ash content, percent by mass, *Max*	0.1	—	**12.1**
v)	α-Naphthol content, percent by mass, *Max*	0.2	C	—
vi)	Naphthalene content, ppm, *Max*	20	D	

[1] In case of disputes, assay by HPLC shall be the referee method.

5 SAMPLING

5.1 The method of preparing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Tests for purity and crystallizing point shall be conducted on the individual samples separately.

5.2.2 Tests for determination of the remaining characteristics, namely, α-naphthol content, insoluble in dilute sodium hydroxide solution, ash content, and naphthalene content shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay and crystallizing point if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (**5.2.2**) the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in col 4 and col 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, ‘pure chemicals’ and HPLC grade water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (ii)]

DETERMENIATION OF *β*-NAPHTHOL CONTENT (PURITY) BY COUPLING VALUE

A-1 APPARATUS

A-1.1 Watch Glass or Paper Boat (Butter Paper/Glossy Paper).

A-1.2 Wash Bottle

A-1.3 Glass Beaker, 500 ml or 1 000 ml.

A-1.4 Glass Rod

A-1.5 Graduated Pipette,10 ml.

A-1.6 Magnate and Magnetic Stirrer

A-2 REAGENTS

A-2.1 Para-nitroanilineDiazo Solution [PNA DIAZO] (Standardized), 0.1 N.

A-2.2 Sodium Acetate (Hydrous)

A-2.3 Starch Iodide Paper (SI paper)

A-2.4 Spot Paper

A-2.5 H-acid Solution, 0.1 percent.

A-2.6 Ice

A-3 PREPARATION OF 0.1 N PNA DIAZO SOLUTION

Weigh accurately approximately 3.520 4 g of para-nitroaniline **(**PNA) (purity 98 percent, *Min*) in 250 ml glass beaker. Add 25 ml of concentrated hydrochloric acid and 25 ml water. Heat to dissolve. Keep the solution under external cooling. Add slowly 27 ml of 1 N sodium nitrite ($NaNO_2$) solution under stirring. Check the SI paper, it should give positive (blue colour spot). Check SI paper test after 15 min, it must be positive. Add sulfamic acid slowly to destroy excess of nitrous acid. Check SI paper, it must be negative (no blue colour spot). Transfer it in a 250 ml volumetric flask and make 250 ml with cold distilled water. Keep the solution in an ice bath.

A-4 STANDARDIZATION OF 0.1 N PNA DIAZO SOLUTION

A-4.1 Weigh accurately 1.0 g of *β*-naphthol (standard) in 500 ml glass beaker. Add approximately 50 ml distilled water. Add 10 percent soda ash solution to dissolve [check brilliant yellow paper (BY paper), it shall be positive]. Add 100 ml distilled water. Add 10 g sodium acetate. Stirrer it on magnetic stirrer for mixing properly, then add ice to cool it. Add 0.1 N PNA diazo solution to it using 10 ml graduated pipette. Check the spot on the spot paper. It should be positive to coupler side (1 percent H-acid solution) and negative to diazo side (that is, for PNA diazo). It shall remain positive to coupler side (0.1 percent H-Acid solution) for 10 min. If not, then add more PNA solution till the spot of reaction show positive to coupler side for 10 min. Note the reading.

A-4.2 End Point

Spot paper positive coupler side (0.1 percent H-acid solution).

Spot paper negative diazo side (that is for PNA diazo).

NOTE — End point remain for 10 min otherwise add more.

A-4.3 Calculation

$$\text{Normality of PNA DIAZO solution} = \frac{99 \text{ x weight of } \beta - \text{Naphthol [standard (STD)] (in g)}}{\text{Reading x 144}}$$

A-5 PROCEDURE

Weigh accurately 1.0 g of sample in paper boat or watch glass. Transfer it to 500 ml or 1 000 ml glass beaker. Add 50 ml caustic solution and dissolve it. Stirrer it on the magnetic stirrer till the solution is clear. Add 10 g sodium acetate. Stirrer it on magnetic stirrer for mixing properly, then add ice to cool it. Add 0.1 N PNA solution to it using 10 ml graduated pipette. Check the spot on the spot paper. It should be positive to coupler side (1 percent H-acid solution) and negative to diazo side (that is, for PNA diazo). It should be remain positive to coupler side (1 percent H-acid solution) for 10 min. If not then add more PNA solution till the spot of reaction show positive to coupler side for 10 min. Note the reading.

A-6 CALCULATION

$$\text{Purity, percent by mass} = \frac{\text{Reading x Normality of PNA Diazo x 144}}{\text{Weight of Sample (in g) x 10}}$$

NOTE — Molecular weight of β-naphthol is 144.

ANNEX B

[*Table* 1, *Sl No.* (iii)]

DETERMENIATION OF *β*-NAPHTHOL CONTENT (PURITY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

B-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 Binary Gradient Liquid Chromatography System, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-3 REAGENT

B-3.1 Acetonitrile, HPLC grade.

B-3.2 Water, HPLC grade.

B-3.3 Disodium Hydrogen Orthophosphate

B-3.4 Ammonium Dihydrogen Orthophosphate

B-3.5 *β*-Naphthol Standard

B-4 STANDARD PREPARATION

Weigh accurately 0.050 0 g standard *β*-naphthol in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

B-5 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

B-6 BUFFER PREPARATION

Take 0.575 0 g ammonium dihydrogen orthophosphate and 0.700 0 g disodium hydrogen orthophosphate in 1 l volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make the total volume with HPLC grade water.

B-7 FLOW RATE, 1.20 ml/min.

B-8 MOBILE PHASE

Time	*Acetonitrile*	*Buffer*
0.01	40	60
10.00	40	60
15.00	70	30
20.00	70	30
20.01	40	60
26.00	Stop	-

B-9 COLUMN OVEN TEMPERATURE, 40 °C.

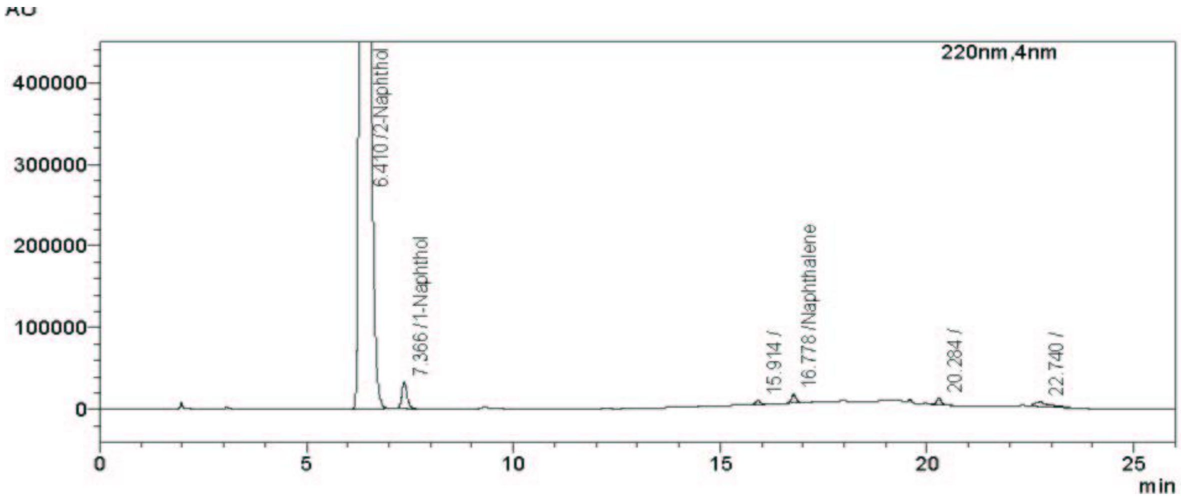
B-10 INJECTION VOLUME, 5 µl.

B-11 RUN TIME, 26 min.

B-12 WAVELENGTH, 220 nm.

B-13 PEAK TIME

β-Naphthol: 6.40 min



B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to β-naphthol on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure β-naphthol.

$$\beta - \text{naphthol, percent} = \frac{A_2 \text{ x } V_1 \text{ x } W_1 \text{ x } B_2}{A_1 \text{ x } V_2 \text{ x } W_2 \text{ x } B_1} \text{ x } 100$$

where

A_1 = area of standard β-naphthol;

V_1 = injection volume of standard β-naphthol;

W_1 = weight of standard β-naphthol;

B_1 = total volume of standard β-naphthol;

A_2 = area of β-naphtholpeak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

ANNEX C

[*Table*1, *Sl No.* (vii)]

DETERMINATION OF α-NAPHTHOL CONTENT IN β-NAPHTHOL

C-1 OUTLINE OF METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

C-2 APPARATUS

C-2.1 Binary Gradient Liquid Chromatography System, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent

C-3 REAGENT

C-3.1 Acetonitrile, HPLC grade.

C-3.2 Water, HPLC grade.

C-3.3 Disodium Hydrogen Orthophosphate

C-3.4 Ammonium Dihydrogen Orthophosphate

C-3.5 α-Naphthol Standard

C-4 STANDARD PREPARATION

Solution A — Weigh accurately 0.050 0 g standard α-naphthol in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

Take 1 ml of solution A in 10 ml volumetric flask and dilute it up to the mark with acetonitrile.

C-5 SAMPLE PREPARATION

Weigh accurately 0.500 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

C-6 BUFFER PREPARATION

Take 0.575 0 g ammonium dihydrogen orthophosphate and 0.700 0 g disodium hydrogen orthophosphate in 1 l volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make the total volume with HPLC grade water.

C-7 FLOW RATE, 1.20 ml/min.

C-8 MOBILE PHASE

Time	*Acetonitrile*	*Buffer*
0.01	40	60
10.00	40	60
15.00	70	30
20.00	70	30
20.01	40	60
26.00	Stop	-

C-9 COLUMN OVEN TEMPERATURE, 40 °C.

C-10 INJECTION VOLUME, 5 µl.

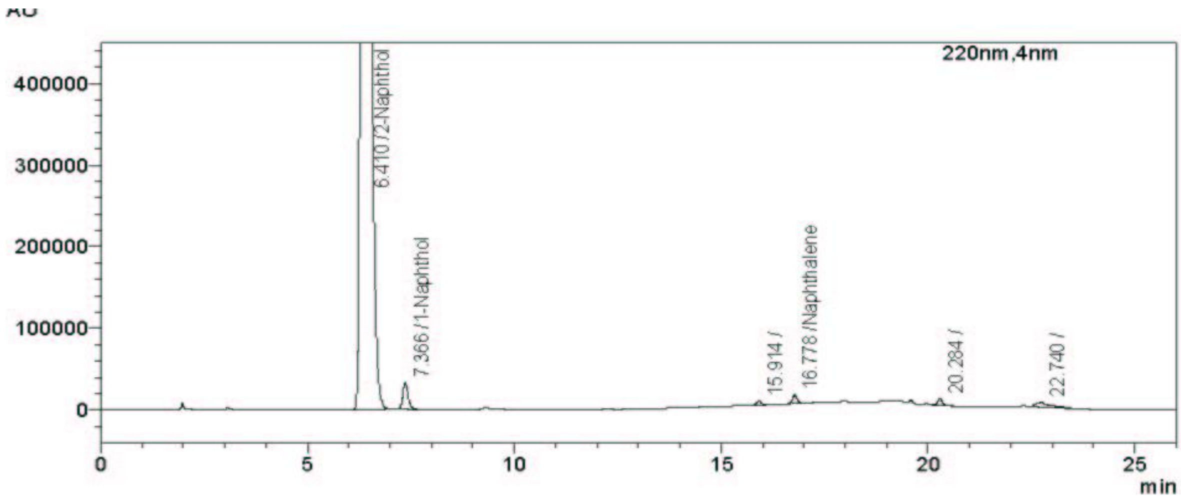
C-11 RUN TIME, 26 min.

C-12 WAVELENGTH, 220 nm.

C-13 PEAK TIME

β-Naphthol: 6.40 min

α-Naphthol: 7.36 min



C-14 CALCULATION

Calculate the peak area of individual constituent pertaining to α-naphthol on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure α-naphthol.

$$\alpha - \text{naphthol, percent} = \frac{A_2 \text{ x } V_1 \text{ x } W_1 \text{ x } B_2}{A_1 \text{ x } V_2 \text{ x } W_2 \text{ x } B_1} \text{ x } 100$$

where

A_1 = area of standard α-naphthol;

V_1 = injection volume of standard α-naphthol;

W_1 = weight of standard α-naphthol;

B_1 = total volume of standard α-naphthol;

A_2 = area of α-naphthol peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample

ANNEX D

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF NAPHTHALENE CONTENT IN β-NAPHTHOL

D-1 APPARATUS

D-1.1 Binary Gradient Liquid Chromatography System, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

D-1.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

D-2 REAGENT

D-2.1 Acetonitrile, HPLC grade.

D-2.2 Water, HPLC grade.

D-2.3 Disodium Hydrogen Orthophosphate

D-2.4 Ammonium Dihydrogen Orthophosphate

D-2.5 Naphthalene Standard

D-3 STANDARD PREPARATION

Solution A — Weigh accurately 0.050 0 g standard naphthalene in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

Take 1 ml of solution A in 50 ml volumetric flask and dilute it up to the mark with acetonitrile.

D-4 SAMPLE PREPARATION

Weigh accurately 0.500 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

D-5 BUFFER PREPARATION

Take 0.575 0 g ammonium dihydrogen orthophosphate and 0.700 0 g disodium hydrogen orthophosphate in 1 l volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make the total volume with HPLC grade water.

D-6 FLOW RATE, 1.20 ml/min.

D-7 MOBILE PHASE

Time	*Acetonitrile*	*Buffer*
0.01	40	60
10.00	40	60
15.00	70	30
20.00	70	30
20.01	40	60
26.00	Stop	-

D-8 COLUMN OVEN TEMPERATURE, 40 °C.

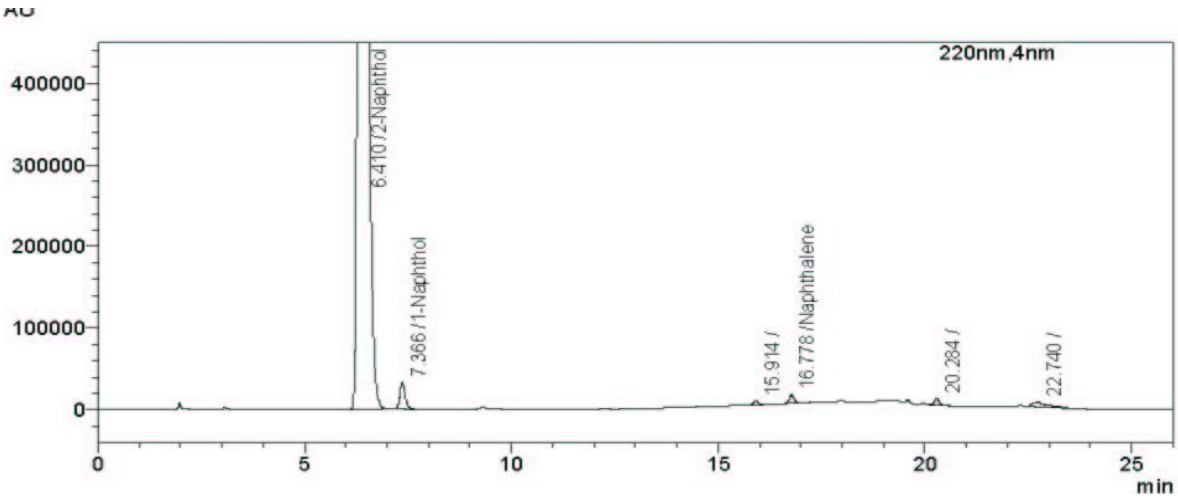
D-9 INJECTION VOLUME, 5 µl.

D-10 RUN TIME, 26 min.

D-11 WAVELENGTH, 220 nm.

D-12 PEAK TIME

β-Naphthol:	6.40 min
α-Naphthol:	7.36 min
Naphthalene:	16.77 min



D-13 CALCULATION

Calculate the peak area of individual constituent pertaining to naphthalene on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure naphthalene.

$$\text{Naphthalene, ppm} = \frac{A_2 \text{ x } V_1 \text{ x } W_1 \text{ x } B_2}{A_1 \text{ x } V_2 \text{ x } W_2 \text{ x } B_1} \text{ x } 10^6$$

where

A_1 = area of standard naphthalene;

V_1 = injection volume of standard naphthalene;

W_1 = weight of standard naphthalene;

B_1 = total volume of standard naphthalene;

A_2 = area of naphthalene peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
Institute of Chemical Technology, Mumbai	Prof Ganapati Subray Shankarling (***Chairperson***)
Aarti Industries Limited, Mumbai	Dr Vaishali Bhandary Dr Sanjeev Kumar Dixit (*Alternate*)
Alkyl Amines Chemicals Limited, Mumbai	Shri S. V. Nikumbhe Shri Kirat Patel (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited, Ankleshwar	Shri Mansukh H Vekaria
Archroma India Private Limited, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited, Gujarat	Dr M. U. Rahman Dr J. G. Desai (*Alternate*)
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Central Revenue Control Laboratory, New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal *(Alternate)*
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Defence Research Development Organization, Ministry of Defence, New Delhi	Dr Vijay Tak
Dystar, Mumbai	Dr Monika Singh
Ecological and Toxicological Association of Dyes, Vadodara	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association, Ahmedabad	Shri Yogesh Parikh Shri Anil M. Jain (*Alternate* I) Shri Haresh Bhuta (*Alternate* II)
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	Shri R. M. Patel Shri C. S. Patel *(Alternate)*
Gujarat Pollution Control Board, Gandhinagar, Ahmedabad	Shri Y. A. Tai

Organization	*Representative(s)*
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Indian Beauty and Hygiene Association, Mumbai	Ms Malathi Narayanan
Indian Chemical Council, Mumbai	Shri P. S. Singh
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Ministry of Environment Forest and Climate Change, New Delhi	Shri N. Subrahmanyam
National Test House, Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
Sudarshan Chemical Industries Limited, Pune	Dr R. Sridharan
The Bombay Textile Research Association, Mumbai	Ms Shital Palaskar
The Dyestuff Manufactures Association of India Office, Mumbai	Shri V. R. Kanetkar
BIS Directorate General	Shrimati Nagamani T, Scientist 'E' and Head (PCD) [Representing Director General (*Ex-officio*)]

Member Secretary
Ms Aditi Choudhary
Scientist 'B', BIS

Working Group for validation of test methods, PCD 26/ WG-1

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Aarti Industries Limited, Mumbai	DR VAISHALI BHANDARY
Atul Limited, Gujarat	DR M. U. RAHMAN
Archroma India Private Limited, Thane	DR RAJESH RAMAMURTHY
Central Revenue Control Laboratory, New Delhi	DR T. A SREENIVASA RAO
Heubach Colour Private Limited, Mumbai	SHRI J. I. SEVAK
Jay Chemicals Industries Private Limited, Ahmedabad	SHRI VILPESH YADAV
NimkarTek Technical Services Pvt Ltd, Thane	SHRI ULLHAS M. NIMKAR

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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